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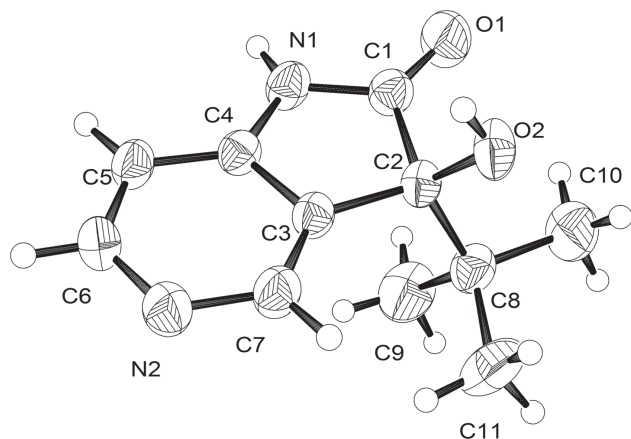
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# Crystal structure of 3-*tert*-butyl-3-hydroxy-1,3-dihydro-2*H*-pyrrolo[3,2-*c*]pyridin-2-one, $C_{11}H_{14}N_2O_2$



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## Abstract

$C_{11}H_{14}N_2O_2$ , orthorhombic,  $P2_12_12_1$  (no. 19),  $a = 7.5411(2)$  Å,  $b = 11.5148(2)$  Å,  $c = 12.5370(2)$  Å,  $V = 1088.64(4)$  Å<sup>3</sup>,  $Z = 4$ ,  $R_{gt}(F) = 0.0301$ ,  $wR_{ref}(F^2) = 0.0826$ ,  $T = 296$  K.

CCDC no.: 1456766

The crystal structure is shown in the figure, Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

## Source of material

The title compound was prepared as previously reported *via* double lithiation of 4-pivaloylaminopyridine with *n*-BuLi (2.1 mole equivalents) in anhydrous THF at 0°C for 3 h followed by

**Table 1:** Data collection and handling.

Crystal:	Colourless, block, size 0.081 × 0.135 × 0.298 mm
Wavelength:	CuKα radiation (1.54184 Å)
μ:	7.17 cm <sup>−1</sup>
Diffractometer, scan mode:	SuperNova, Dual, Cu at zero, Atlas, ω scans
2θ <sub>max</sub> :	148.2°
$N(hkl)_{measured}$ , $N(hkl)_{unique}$ :	3668, 2123
$N(param)_{refined}$ :	141
Programs:	CrysAlis <sup>PRO</sup> [5], SHELX [6], WinGX [7], ChemDraw [8]

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>iso</sub>
H(5)	4a	0.1302	1.0553	0.0934	0.0480
H(6)	4a	0.3050	1.0206	−0.0539	0.0520
H(7)	4a	0.3790	0.6972	0.02510	0.0500
H(9A)	4a	0.5447	0.7417	0.3790	0.1000
H(9B)	4a	0.3600	0.8025	0.3922	0.1000
H(9C)	4a	0.4705	0.8209	0.2877	0.1000
H(10A)	4a	0.3884	0.5571	0.4235	0.0970
H(10B)	4a	0.2357	0.5112	0.3498	0.0970
H(10C)	4a	0.2024	0.6179	0.4249	0.0970
H(11A)	4a	0.5359	0.6524	0.1737	0.1030
H(11B)	4a	0.4211	0.5392	0.1812	0.1030
H(11C)	4a	0.5708	0.5625	0.2653	0.1030
H(1)	4a	0.0055	0.9476	0.2712	0.0480
H(2)	4a	0.0242	0.6203	0.1404	0.0660

a reaction with carbon monoxide. The crude product obtained was purified by crystallization from ethyl acetate to give the title compound in 65% yield as colourless crystals [1].

## Discussion

Synthesis of azaindoles is of great interest due to their growing biological applications [2]. The asymmetric unit consists of one molecule of  $C_{11}H_{14}N_2O_2$ . In the crystal structure, the hydroxyl group accepts one N–H···O hydrogen bond from a neighbouring molecule (N1–H1···O2 angle = 169.63°,

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**Table 3:** Fractional atomic coordinate and displacement parameters (Å<sup>2</sup>).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>11</sub>	<i>U</i> <sub>22</sub>	<i>U</i> <sub>33</sub>	<i>U</i> <sub>12</sub>	<i>U</i> <sub>13</sub>	<i>U</i> <sub>23</sub>
C(1)	4a	0.0581(3)	0.7795(2)	0.2890(1)	0.0419(9)	0.0402(9)	0.0364(8)	0.0029(8)	0.0009(8)	0.0024(8)
C(2)	4a	0.1868(2)	0.7014(1)	0.2231(1)	0.0392(9)	0.0281(8)	0.0367(8)	−0.0005(7)	−0.0012(7)	0.0017(6)
C(3)	4a	0.2355(2)	0.7836(1)	0.1328(1)	0.0358(8)	0.0278(7)	0.0363(8)	0.0001(7)	−0.0026(7)	0.0000(7)
C(4)	4a	0.1578(2)	0.8916(1)	0.1517(1)	0.0356(8)	0.0300(8)	0.0354(8)	0.0007(7)	−0.0030(7)	−0.0021(6)
C(5)	4a	0.1812(3)	0.9829(2)	0.0818(2)	0.0428(9)	0.0275(8)	0.049(1)	0.0017(7)	−0.0024(9)	0.0034(7)
C(6)	4a	0.2847(3)	0.9599(2)	−0.0064(2)	0.046(1)	0.0376(9)	0.047(1)	−0.0022(8)	0.0019(9)	0.0118(8)
C(7)	4a	0.3312(3)	0.7698(2)	0.0404(2)	0.048(1)	0.0336(9)	0.0426(9)	0.0069(8)	0.0063(9)	0.0004(7)
C(8)	4a	0.3488(3)	0.6613(2)	0.2921(2)	0.0434(9)	0.0393(9)	0.044(1)	0.0023(8)	−0.0085(8)	0.0025(7)
C(9)	4a	0.4394(4)	0.7663(2)	0.3424(2)	0.062(1)	0.060(1)	0.078(2)	−0.004(1)	−0.027(1)	−0.004(1)
C(10)	4a	0.2882(4)	0.5793(2)	0.3808(2)	0.062(1)	0.071(2)	0.061(1)	0.000(1)	−0.017(1)	0.027(1)
C(11)	4a	0.4814(4)	0.5980(3)	0.2216(2)	0.067(2)	0.073(2)	0.067(2)	0.034(1)	−0.006(1)	0.004(1)
N(1)	4a	0.0594(2)	0.8886(1)	0.2445(1)	0.0487(8)	0.0339(7)	0.0385(8)	0.0088(7)	0.0043(7)	−0.0017(6)
N(2)	4a	0.3582(2)	0.8572(2)	−0.0288(1)	0.0509(9)	0.0437(9)	0.0443(9)	0.0044(7)	0.0099(8)	0.0072(7)
O(1)	4a	−0.0295(2)	0.7513(1)	0.3652(1)	0.071(1)	0.060(1)	0.0525(8)	0.0116(8)	0.0230(8)	0.0138(7)
O(2)	4a	0.0957(2)	0.6015(1)	0.1865(1)	0.0555(8)	0.0306(6)	0.0455(7)	−0.0087(6)	−0.0133(6)	0.0068(5)

N1···O2 distance = 2.851 Å) and donates one O—H···N bond to another molecule (O2—H2···N2 angle = 172.48°, O2···N2 distance = 2.710 Å) leading to a 3-D network. An extremely weak C—H···O contact is also observed (C5—H5···O1 angle = 173.13°, C···O distance = 3.362 Å) resulting in R<sup>2</sup><sub>2</sub>(9) graph set descriptor for this motif [3] between two molecules. The shortest C—H···O contact in the related 3-isopropyl-1-methyl-1,3-dihydro-2*H*-pyrrolo[3,2-*c*]pyridin-2-one [4] involves a tertiary C atom (C—H···O angle = 144.18° and C···O distance = 3.292 Å).

### Experimental details

H atoms were placed in calculated positions and refined using a riding model, with *U*<sub>iso</sub>(H) set to 1.2*U*<sub>eq</sub>(C) and C—H and N—H distance of 0.93 and 0.86 Å respectively. The exceptions were the methyl (C—H = 0.96 Å) and hydroxyl (O—H = 0.86 Å) groups which were allowed to rotate around the C—C bond (HFIX 137 in SHELX [6]) and C—O bond (HFIX 147), with *U*<sub>iso</sub>(H) set to 1.5*U*<sub>eq</sub>(C/O).

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